J130 Report No. 224

FINAL REPORT

HIGH TEMPERATURE DIELECTROMETER FOR CENTIMETER AND MILLIMETER WAVES

WORK PERFORMED FOR
AIR RESEARCH AND DEVELOPMENT COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO
UNDER U. S. AIR FORCE CONTRACT
AF 33(616)-3256

This report is not to be announced or distributed automatically, in accordance with AFR 205-43, Paragraph 6d.

MICROWAVE RADIATION COMPANY, INC.
19223 South Hamilton Street
Gardena, California

CONTENTS

ABSTRACT

This report is a summary of the design and development program leading to the construction of a High Temperature Dielectrometer for Centimeter and Millimeter Waves. The logical design of the instrument is the same as that of the Dielectrometer constructed on Contract AF33(616)-2889. Design problems discussed in this report are concerned with materials and methods for use at high temperature.

I. PURPOSE OF THE CONTRACT

The purpose of this contract is to develop, design and fabricate a high temperature surface wave transmission line which shall be capable of utilizing all the electronic and control components developed for the 600°C dielectrometer under Contract AF33(616)-2889 and thus extend the range of the initial dielectrometer to 1,000°C.

The requirements on performance of the dielectrometer when fitted with this high temperature surface wave transmission line include:

1. Temperature Range: Ambient to 1,000°C.
2. Range in Dielectric Constant: 1 to 50.
3. Range in Loss Tangent: 0.0001 to 0.5.
4. Precision of Dielectric Constant Measurement: ± .01.
5. Accuracy of Dielectric Constant Measurement: ± .05.
6. Precision of Loss Tangent Measurement: ± 1%.
7. Accuracy of Loss Tangent Measurement: ± 5%.
8. Sample Size - larger than 1 x 1 x 1/8 inches, but smaller than 10 x 8 x 1 inches.
9. Operational suitability - The technique must be capable of convenient determination of the properties of the dielectric and require a minimum time for the taking of measurements

and a minimum amount of computation in relating measured quantities to the properties of the dielectric sample.

10. An operational life of at least 100 hours.

II. FACTUAL DATA AND DISCUSSION

A. INTRODUCTION AND DISCUSSION

This report covers the design and construction of an instrument to be used in conjunction with the "Dielectrometer for Millimeter Waves" developed under Contract AF33(616)-2889. The subject instrument is identical in operation and interchangeable with the basic unit constructed on the earlier contract. The only differences between the two units are their temperature ranges, construction materials and minor changes in mechanical design.

The dielectric constant and loss tangent of dielectric materials is determined by measuring the standing wave on a shorted length of surface wave transmission line. The surface wave transmission line is formed from the dielectric sample (a sheet approximately 7 in. x 8 in.) and a flat metallic surface. One end of the dielectric sheet is connected through suitable transitions to a signal source. A shorting plate is located at the other end of the dielectric sheet. The standing wave on the surface wave transmission line formed by the sample is sampled by means of a small slot in the metallic surface. The central section of the metallic surface, in which the probe slot is located, is free to translate along the direction of propagation of the surface wave. The probe slot is connected by waveguide to a suitable receiver. A detailed description and analysis of the principles of operation of the dielectrometer is contained in the various progress reports published by the Microwave Radiation Company, Inc., in connection with Contract AF33(616)-2889 and in the operating instructions for the instrument.

The dielectrometer to be discussed in this report consists of a massive metal stand on which the temperature controls, a measuring microscope, a cable drive unit and an oven unit (containing the shorted surface wave transmission line) are mounted. The section of the base block of the surface wave transmission line which contains the probe slot is connected by means of a flexible cable to the cable drive mechanism. The probe slot may be translated over a distance of about 2 inches by turning a large handwheel on the cable drive unit.

The translatory motion of the probe slot is measured with a measuring microscope located outside the oven unit. A fiduciary mark attached to the section carrying the probe slot is observed through a window in the oven wall and its motion measured with the measuring microscope.

The temperature controls consist of a Leeds and Northrup Type G temperature recorder and a Series 60 controller. The controls are connected to a large solenoid contactor which pulses the electrical heating elements on and off. The heating elements are of the resistive type and are located in the oven unit. Maximum operating temperature for the unit is approximately 1000 degrees centigrade. The temperature is measured with a thermocouple junction located in the oven.

B. MATERIALS INVESTIGATION

Because the electrical design of the surface wave dielectrometer had already been accomplished, the principal factor controlling the design of the instrument was the choice of materials to withstand the extreme temperature requirements.

The surfaces of the input transition, base block and shorting plate of the surface wave transmission line must be a metal with good electrical conductivity. However, these surfaces do not have to be more than one thousandth of an inch thick. Thus, the possibility exists of making the parts of any material so long that it can be covered with a metallic film.

The only practical metals with good conductivity which are useful at 1000 degrees centigrade are gold and platinum. Platinum is desirable from the standpoint of its excellent refractory properties, but gold is a much better conductor of electricity. However, since the required 1000 degrees centigrade is just a few degrees below the melting point of gold, the use of gold presents difficulties.

The use of a ceramic material for the base block and slider appears to present many advantages over the use of metals. These advantages are thermal stability and low thermal expansion and the fact that centuries old techniques are available for firing gold and platinum coatings on ceramics. The disadvantages in the use of ceramics lie in the difficulty of manufacture and low heat conductivity.

The size of the base block and slider (about 9 x 12 x 3 inches) is large compared to parts that are usually made from ceramics. Two other factors make the manufacture from ceramic materials somewhat of a development project: (1) It is necessary to maintain sharp edges on the surfaces where the base block and slider meet; (2) the thicknesses of the parts are large compared to those usually encountered in precision molded ceramics. Thus it is difficult to estimate in advance the shrinking and warping of the parts that might occur in firing.

The parts can probably be molded from a high strength ceramic such as one of the steatites and finished ground to a sharp enough edge. However, considerable experimentation would be required to design the molds and firing processes so as to maintain dimensions after firing that would not require an excessive amount of finish grinding. Because high pressure molds and the grinding of ceramics are both quite costly, the use of molded ceramics seems inappropriate to the manufacture of a small number of parts that are as difficult to fabricate as the block and slider.

One ceramic material, Grade A Lava (a natural stone marketed by the American Lava Corporation), does not have some of the disadvantages listed above. This material is readily machined in the unfired state and after firing becomes a hard, relatively dense ceramic with a softening point at 2900 degrees F. Because the material is relatively inexpensive and does not require molding, Lava is an excellent material for experimental fabrication. It has been found that it is entirely feasible to fabricate the material with sharp edges, or to grind to a sharp edge with metal working tools after firing. However, such sharp edges are so brittle and easily chipped as to make the use of the material impractical for the base block. In addition, it is difficult to fire pieces as large as those required for the base block without cracking.

Another approach to the use of ceramic materials for the surface wave line is to make base block and slider out of metal and to coat those surfaces that must be kept free of corrosion and scaling with a thin ceramic layer. The principal disadvantages to this process are again connected with the sharp edges required. When a flat surface is coated, the ceramic tends to build up and round over a sharp edge. Thus, finish grinding is required after firing to maintain a flat surface with a reasonably sharp edge. No combination of coatings and/or grinding techniques were discovered which would insure the fabrication of the sharp edges without chipping.

Only two metal alloys appear to be readily available and appropriate for use in fabrication of the surface wave line. The two alloys are Inconnel X and Hastelloy X, which are proprietary materials of the International Nickle Company and Haynes Stellite Company, respectively. Other metals which have sufficient strength and resistance to scale and corrosion at 1000 degrees centigrade, are either not commerically available in the forms required, or are more difficult to fabricate by standard techniques.

Hastelloy X and Inconnel X are not normally cast to shape and are supplied by the mills in the form of sheets, bars and plates. Both of these alloys are difficult to machine even in the fully annealed state. However, they can be worked with ordinary high speed tools if the tools are kept extremely sharp and if the machine operator is careful to maintain a uniform cutting speed.

An experimental investigation was made to determine the feasibility of firing gold and platinum pastes and Lava, Hastelloy X and Inconnel X. From the standpoint of electrical conductivity after firing and mechanical strength, the most successful pastes of those tried were Gold Paste No. 601-FM and Platinum Paste No. 6082, both of which are standard products of the Hanovia Chemical Co. The gold paste yields durable films on Lava which withstand temperatures almost as high as the melting point of the gold when fired at temperature above 1400 degrees F. Fired gold coatings on the metals were found to be unsatisfactory for use at temperatures above about 800°C - probably due to diffusion of the gold into the base metal. The platinum paste gave good results on either metal, or ceramic, at temperatures in excess of 1000°C when fired on at a temperature of about 850°C. Both the pastes are very easy to apply; they are supplied in a form that can be thinned with turpentine and applied with a brush. The paste is applied and slowly raised to a temperature sufficient to reduce the metallic salts and drive off the solvents, leaving a pure metallic coating. When the metallic pastes are fired on ceramic, a modification of the ceramic surface occurs in which the metallic atoms are included in the crystalline structure of the ceramic. The resulting boundary might be loosely described as a gradual transition from ceramic to metal bearing ceramic to pure metal. Thus, a very strong bond is formed between the metallic coating and the ceramic. Little investigation has been made of the firing of noble metal films on metals, but it is assumed that the process is similar to that described for ceramics. One difference seems apparent, however, and that is that the noble metal will diffuse into another metal more rapidly than into ceramics at high temperatures. Considerably more care is required in surface preparation and paste application in order to obtain satisfactory coatings on metal. In applying gold and platinum to Hastelloy X, the gold was found to be considerably easier to use - probably because the firing temperatures did not approach the softening points of either the platinum or the base metal, but did approach the softening point of gold.

C. DESIGN AND CONSTRUCTION

The design and construction of the High Temperature Dielectrometer is almost identical to the instrument previously constructed for use at temperatures up to 600°C. The differences are materials, some dimensional changes required by the use of different materials, and a modification of the stand, so as to provide more rigid support for the input feed horn.

The oven enclosure is identical to that of the previous instrument, except that the insulation thickness has been increased and a different insulation material used. The insulation is a sandwich, with "Asbestolux" insulating board on the inner and outer surfaces, separated by "Kaylo 1800" insulating material. Manufacturer's specifications state that the insulating material will withstand temperatures of 2300°F.

The surface wave transmission line and its input transition is constructed of Hastelloy X, for reasons stated in the previous section. Little attention was given to the use of Inconnel material, since the forms required were not available at the time of construction, due to a prolonged labor dispute at the International Nickle Co. The Hastelloy was obtained from the manufacturer, in the form of one inch thick plate cut into bars of the approximate shapes to be used.

An advantage, not previously mentioned, in the use of metal for the base of the surface wave line is that it provides an excellent means for heating the sample. The heating elements are imbedded in the base block and, thus, the block and sample are brought to a uniform temperature. The design of the heating elements is simplified by the fact that the outer sheath of the element need not be hotter than the desired sample temperature. No safety devices are required to reduce the heater current at high temperatures. The heaters used are of the tubular type constructed for this application by the Industrial Heating Co. The heaters consist of a metal sheath approximately 16 in. long and 5/8 in.

in diameter, ceramic insulation and a Kanthal wire resistive element. Eight such heaters are employed.

The base block is fabricated from five ground blocks of Hastelloy, which are assembled with locating pins and screws. The heating element holes are obtained by machining semi-circular grooves in mating surfaces of the assembled block. The blocks are then assembled and the holes formed from pairs of matching grooves, are reamed to fit the heating element sheath. This procedure was used to avoid having to drill deep (about 13 inches) holes in the Hastelloy - a very difficult task. The slider does not contain heating elements but is heated by conduction from the base block.

After assembly, the base block and slider are carefully ground so that the slider ways are parallel to the surface of the base block, within a tolerance of .0002 inches. The slider face is ground flat, parallel and to a thickness that locates the upper face of the slider between .0001 and .0003 inches below the surface of the base block.

The input transition horn and the output waveguide are fabricated from several pieces that are brazed together with a nickle braze alloy (Handy and Harmon "High Temp." No. 91). This brazing material forms a good mechanical joint that maintains its strength at temperatures well above 1000°C. Because this brazing alloy is quite new, little or no information is available concerning the best techniques for brazing Hastelloy. In brazing the dielectrometer parts, it was found that a good bond was nearly always obtained. However, considerable difficulty was encountered with warping and the destruction of surface finish caused by the brazing material running out of the seams. The input and output waveguide sections leading through the oven wall are made of Grade A Lava. The parts are machined in two halves, fired, finish ground, bonded together with refractory cement and the inside surfaces are platinum coated by firing of several coats of platinum paste.

All of the surfaces of the metallic parts in the oven which are electrically important are coated by firing on of two or more layers of platinum.

D. CONCLUSIONS

Construction of the High Temperature Dielectrometer has demonstrated that materials and techniques are available for successful design of such instruments for use at very high temperatures. However, because some of the fabrication techniques are somewhat experimental and because nearly all the useful materials are difficult to fabricate, the 1000° instrument is considerably more costly to manufacture than an instrument designed for somewhat lower temperatures.